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Petroleum and related products — Precision of measurement methods and results —

Part 4:
Use of statistical control charts to validate 'in-statistical-control' status for the execution of a standard test method in a single laboratory

Produits pétroliers et connexes — Fidélité des méthodes de mesure et de leurs résultats —

Partie 4: Utilisation de cartes de contrôle statistique pour valider l'état 'sous maîtrise statistique' pour l'exécution d'une méthode d'essai normalisée dans un seul laboratoire

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Foreword

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The procedures used to develop this document and those intended for its further maintenance are described in the ISO/IEC Directives, Part 1. In particular, the different approval criteria needed for the different types of ISO documents should be noted. This document was drafted in accordance with the editorial rules of the ISO/IEC Directives, Part 2 (see www.iso.org/directives).

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For an explanation of the voluntary nature of standards, the meaning of ISO specific terms and expressions related to conformity assessment, as well as information about ISO's adherence to the World Trade Organization (WTO) principles in the Technical Barriers to Trade (TBT), see www.iso.org/iso/foreword.html.

This document was prepared by Technical Committee ISO/TC 28, *Petroleum and related products, fuels and lubricants from natural or synthetic sources*, in collaboration with the European Committee for Standardization (CEN) Technical Committee CEN/TC 19, *Gaseous and liquid fuels, lubricants and related products of petroleum, synthetic and biological origin*, in accordance with the Agreement on technical cooperation between ISO and CEN (Vienna Agreement).

A list of all parts in the ISO 4259 series can be found on the ISO website.

Any feedback or questions on this document should be directed to the user's national standards body. A complete listing of these bodies can be found at www.iso.org/members.html.

Introduction

In the current global business environment, measurement data ‘trustworthiness’ is a key business driver and an implicit expectation from customers and regulatory entities. Data trustworthiness means the data quality meets expectations and is ‘fit-for-use’. Trustworthy data can only be produced by measurement systems that are demonstrated to be stable and are under common cause variation only.

This document describes the applications of specific statistical control charts selected from those that are widely used by the manufacturing sector for the purpose of monitoring and demonstrating the in-statistical-control status of a laboratory in the execution of a standardized test method to produce trustworthy data.

In ISO 4259-2[9], the requirement for assessment of product quality conformance to specification, is to be interpreted that each laboratory’s test result is obtained from a test method that is in-statistical-control in terms of precision and bias, to be substantiated by in-house statistical quality control (SQC) charts or other equivalent statistical techniques. While in-house techniques are used by many laboratories for test method quality assurance, standardization on how to establish in-statistical-control is necessary to ensure consistency in application of ISO 4259-2[9]. Addressing the aforementioned necessity is the motivation of this document, which is based on ASTM D6299[1].

Petroleum and related products — Precision of measurement methods and results —

Part 4: Use of statistical control charts to validate 'in-statistical-control' status for the execution of a standard test method in a single laboratory

1 Scope

This document specifies the process and methodology for the construction, operation, and maintenance of statistical control charts to assess if a laboratory's execution of a standard test method is in-statistical-control and how to establish and validate the 'in-statistical-control' status.

It specifies control charts that are most appropriate for ISO/TC 28 test methods where the dominant common cause variation is associated with the long term, multiple operator conditions. The control charts specified for determination of in-statistical-control are: individual (I), moving range of 2 (MR_2), and either the exponentially weighted moving average (EWMA) or zone-based run rules [similar to Western Electric (WE) run rules[3]] as sensitivity enhancement strategy to support the I-chart.

The procedures in this document have been primarily designed for numerical results obtained from testing of control samples prepared from a homogenous source of petroleum and related products in a manner that preserves the homogeneity of properties of interest between control samples. If the test method permits, a certified reference material (CRM) sample is used as a control sample provided the sample composition is representative of the material being tested and is not a pure compound; if this is done then the laboratory best establishes its own mean for the CRM sample.

This document is applicable to properties of interest that are (known to be) stable over time, and for data sets with sufficient resolution to support validation of the assumption that the data distribution can be approximately represented by the normal (Gaussian) model. Mitigating strategies are suggested for situations where the assumption cannot be validated.

2 Normative references

The following documents are referred to in the text in such a way that some of their content support requirements of this document. For dated references, only the edition cited applies. For undated references, the latest edition of the referenced document (including any amendments) applies.

ISO 4259-1:2017, *Petroleum and related products — Precision of measurement methods and results — Part 1: Determination of precision data in relation to methods of test*

3 Terms, definitions, symbols and abbreviated terms

For the purposes of this document, the terms and definitions given in ISO 4259-1 and the following apply.

ISO and IEC maintain terminology databases for use in standardization at the following addresses:

— ISO Online browsing platform: available at https://www.iso.org/obp

— IEC Electropedia: available at https://www.electropedia.org/

3.1 Specific terms and definitions

3.1.1
common cause
factors that contribute to *common cause variation* (3.1.2)

Note 1 to entry: See Figure 1 for illustration.

3.1.2
common cause variation
variation amongst results collected under *site precision conditions* (3.1.4) from repeated execution of a test method on the same test material attributable to known, unknown, or unknowable factors that are intentionally not or cannot be rigidly controlled as part of the normal and correct execution of all aspects of the test method

3.1.3
in-statistical-control
situation wherein the test results produced by the user on control samples are reasonably consistent with expectation over time with common cause variation scattered around a stable expected centre

3.1.4
site precision conditions
conditions under which single test results are obtained in time intervals, separated by at least 8 h, by the testing population in a single laboratory executing the same test method using the same apparatus on test specimens taken at random from the same material over the normal daily operating envelope

3.1.5
quality control sample
QC sample
specimen taken from a stable and homogeneous material with composition and properties similar to sample normally tested by the laboratory, prepared in a manner that preserves the homogeneity of property of interest between test specimens, stored in a manner that preserves the properties of interest over time, and available in sufficient quantity for repeated long-term testing

3.2 Symbols and abbreviated terms

AD	Anderson Darling
ARV	assigned reference value
CRM	certified reference material
EWMA	exponentially weighted moving average
GESD	generalized extreme studentized deviation
MR_2	moving range of two
$\overline{MR}_{\text{known}}$	moving range average associated with s_{known}
PT	proficiency testing
QC	quality control
q-q	quantile-quantile, term used to describe the plot type comparing the z-score of a data point with is numerical value
s_{known} [a]	statistically pooled standard deviation obtained using final achieved standard deviations from a group of retired control charts where all the final achieved control chart averages are determined to be not statistically significantly different using the appropriate clause(s) in stage 1

[a] The range spanned by the final achieved control chart averages are referred to as the working range associated with s_{known}. See concept illustrated in Table 1.

Table 1 — Statistically pooled standard deviation concept

Material	Property (unit)	s_{known}	*df*	Working range	$\overline{MR}_{known}$
summer gasoline	vapour pressure (kpa)	0,55	60	49,85 to 50,68	0,62
winter gasoline	vapour pressure (kpa)	0,83	85	104,67 to 105,91	0,93

4 Statistical control in the execution of a standard test method by a laboratory

4.1 General

The execution of a standard test method by a laboratory is in this document considered as the execution of a series of inter-connected work processes. Each work process is subject to variation caused by known, unknown, or sometimes unknowable causes that are inherent to a process over long time horizon such that every outcome of the process is affected. These causes are referred to as common causes. The effect on the final process outcome due to common causes are referred to as common cause variation.

Common causes for variation can be grouped into 5 categories (environment, operator, equipment, procedure and reagent material) using a technique known as a fishbone diagram. Due to common cause variation, repeated execution of the same test method on the same material over a long-time horizon yields results that are not numerically identical. This effect is illustrated in Figure 1.

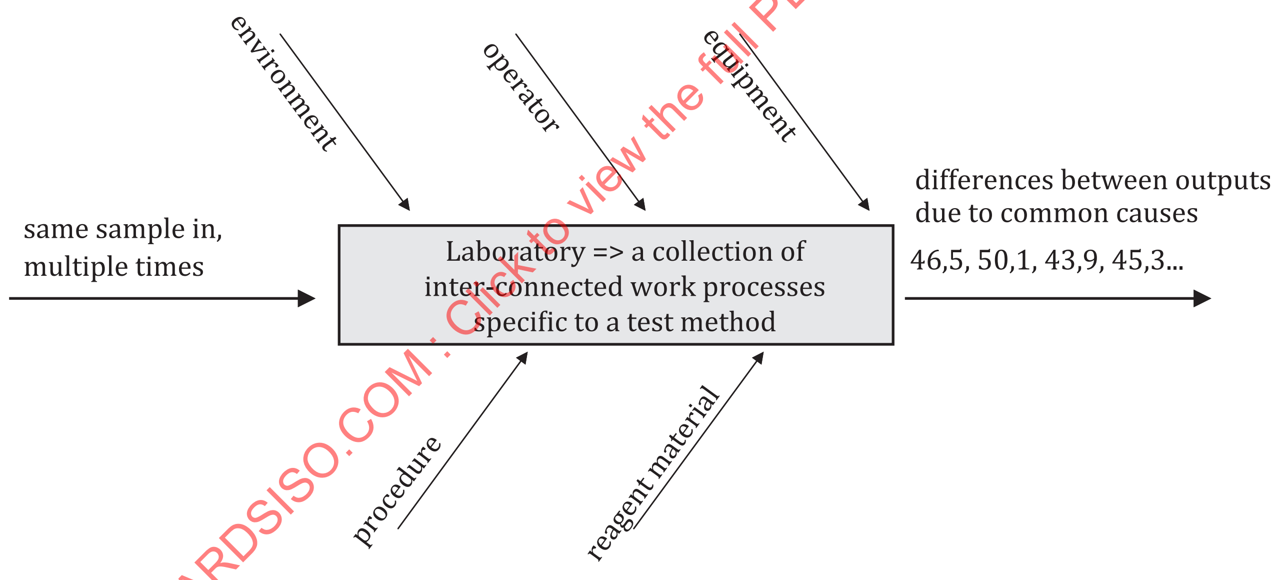


Figure 1 — Fishbone diagram representation of common cause variation in the execution of a test method

The complete process associated with the execution of the specific test method is said to be 'in-statistical-control' if the process outcomes (test results) from repeated analysis of QC samples prepared from the same material are reasonably consistent with expectation over time, with random variation scattered around a stable centre due to common causes only.

To determine 'in-statistical-control', a multi-step and integrated work process involving use of statistical control charts and a QC sample is required.

NOTE For simplicity, the word 'statistical' will be omitted and the term referred to as 'control charts' throughout the rest of this document.

4.2 Control chart description

4.2.1 General

Control charts appropriate for most petroleum industry test methods that yield numeric results are the individual (I) chart, and the moving range of 2 (MR_2) chart.

NOTE 1 The I-chart is also known as the X-chart, or the Shewhart chart [2].

NOTE 2 For simplicity, the MR_2 chart is referred to as the moving range (MR) chart throughout the rest of this document.

4.2.2 I- and MR-charts

The I-chart is a graphic display of individual QC sample test results (X) collected under site precision conditions, plotted in chronological order, overlaid with a centre line, lower and upper decision limits that require action if exceeded. These limits are herein referred to as I-chart lower and upper control limits (LCL_X, UCL_X). The primary purpose of the I-chart is to monitor process centre stability over time.

NOTE Since the primary interest is to monitor the stability and common cause variation of the test process under site precision conditions over a long time horizon, replicate analysis obtained under repeatability conditions (see ISO 4259-1) does not contribute towards this objective, as the variation due to common causes of interest is not contained in replicate results collected under repeatability conditions.

The MR-chart is the successive difference (with no arithmetic sign) of two individual results in the I-chart, plotted in chronological order, also overlaid with a centre line and an upper decision limit for action, herein referred to as MR-chart upper control limit (UCL_MR). The primary purpose of the MR-chart is to monitor common cause variation stability between successive QC sample results over time.

The decision limits for action for both charts as well as the conditions requiring action for the strategies in 4.2.3 are based on a very low theoretical probability (<0,3 %) of “action required” decision for a process that is in-statistical-control, using the Normal distribution (I-chart) and W distribution (MR-chart) as the reference statistical models. Hence, these limits represent the expectation limits for the process outcome if it is in-statistical-control.

4.2.3 I-chart sensitivity enhancement strategy

As a direct consequence of setting the action limits for the I-chart based on a low probability of exceedance for a process that is in-statistical-control, these limits are not sensitive to detection of small changes in the process centre. It is therefore necessary to support I-chart with additional sensitivity enhancement strategies to overcome this shortcoming.

This document requires use of one of the following strategies in conjunction with the I-chart:

a) Strategy 1: Zone-based run rules. Action is required if any of the following run rule conditions is present. For definitions of zones see 4.3.2:

— two out of three consecutive individual results in Zone A on one side of the centre line;

— four out of five consecutive individual results beyond Zone C on one side of the centre line;

— nine consecutive individual results on the same side of the centre line (above or below);

or,

b) Strategy 2: Exponentially weighted moving average (EWMA). Action is required if either one of the following conditions occur:

— any exceedance of the EWMA action limits;

— nine consecutive individual results on the same side of the centre line (above or below).

The EWMA is a ‘time-weighted moving average’ calculated using all data points up to the most current one, the weighting of each datum reduced with age exponentially. The rate of this weight decay is controlled by λ. It is re-calculated with the arrival of each new datum and is judged against its own action limits (herein referred to as the EWMA-action limits). An EWMA with λ = 0,4 has similar detection power as Strategy 1[4].

Use of Strategy 2 is recommended due to the ease of implementation and lower expected false alarm rate than Strategy 1.

4.2.4 In-statistical-control conditions

The complete process associated with the execution of a test method is deemed to be in-statistical-control if all of the following conditions are met:

a) all individual control sample results are within the I-chart action limits,

b) less than five out of 12 successive MR results exceed MR-chart upper control limit, and

c) no action required for the sensitivity enhancement chosen (Strategy 1 or 2).

4.3 Control chart work process

4.3.1 General

To determine if the complete process associated with the execution of a test method is in-statistical-control, a two-stage multi-step work process involving use of control charts and QC material is specified.

Stage 1 comprises visual and statistical assessment of initial test results for a new batch of QC material plotted in a chronological order (known as a run chart). This is followed by construction of the I-chart and MR-chart using these results by overlaying the mean and the action limits onto the respective charts. The action limits represent the boundaries within which the current and future test results and MR for this QC material are expected to lie, on the assumption that the process is in-statistical-control and the QC material remains unchanged. The control charts (I and MR) constructed in Stage 1 are deployed for Stage 2 if all in-statistical-control conditions (see 4.2.4) are met.

Stage 2 comprises of two modes, operation and maintenance. Under 'operation', future test results (for the QC material tested in Stage 1) as they arrive in chronological order are compared against the established action limits and chosen enhancement strategy in Stage 1. Under 'maintenance', the statistics used in the computation of the control chart action limits from Stage 1 are re-assessed periodically using newly accrued in-statistical-control results and updated as appropriate.

For this practice, the root-mean-square technique is used to compute the sample standard deviation statistic, *s*.

4.3.2 Stage 1 of control chart work process

The primary objective of Stage 1 is to establish initial means and action limits for the I, MR control charts and implement the chosen enhancement strategy (see 4.2.3) for a specific batch of QC material using chronologically obtained data and the normal distribution as the reference model. Figure 2 is a flow chart of the 15-step process defined in this subclause. The main steps are:

1) Prepare multiple QC samples from a stable and homogeneous material with composition and properties similar to samples normally tested by the laboratory.

2) Collect a minimum of 20 QC sample test results under site precision conditions.

3) Plot the individual results chronologically (this is called a run chart) and study the plot for any visually discernible transcription errors. Correct or discard obvious transcription errors.

4) Construct a quantile-quantile (q-q) plot (as shown in Annex A) and compute the Anderson-Darling (AD) statistic. Examine this plot in conjunction with the data for variation sufficiency. Proper application of the control charts in this document require at least 6 unique values to provide sufficient observable common cause variation. Insufficient variation in the data set will manifest into the following two outcomes:

   a) q-q plot shows several distinct horizontal data clusters where each horizontal cluster represent numerically identical data (see Clause 5),

   b) AD value exceeds the 0,01 sig. level of 1,0 by a large margin.

   If the total number of unique values is less than six, proceed directly to Clause 5 for guidance.

5) Perform a formal statistical assessment for outliers using generalized extreme studentized deviation (GESD) technique for outliers similar to ISO 4259-1 (or refer to ASTM D7915[5]). The recommended maximum number of outliers for 20 to 25 observations is 3 at 0,01 significance.

   Reject identified outliers, obtain replacement results and repeat from step 4).

   While not always possible, it is recommended that rejection of outliers be justified by corresponding root cause(s).

6) Confirm the goodness-of-fit of the normal distribution using the Anderson-Darling (AD) statistic computed from at least 20 non-outlier results.

   — If AD is less than 1,0, continue to step 7);

   — if AD is between 1,0 and 1,5, proceed to Clause 5 for guidance;

   — if AD is greater than 1,5, do not proceed with this document as this is strong statistical evidence that either the system is not in-statistical-control, or, the process results distribution is severely non-normal.

7) Compute the average ($\bar{x}_{\text{stage1}}$) and standard deviation (s_{stage1}) statistics using the non-rejected results from step 5).

8) Assess if additional data from historic results for this test method can be used to improve the estimate of s_{known}, this should be done using an F-test at the 0,025 significance level with the numerically larger number in the numerator. The reproducibility statement function determines how this should be done:

   a) For methods with a constant published reproducibility: use the F-test assess if s_{stage1} is statistically indistinguishable versus s_{known}.

   b) For methods with a non-constant published reproducibility: calculate the reproducibility at the $\bar{x}_{\text{stage1}}$ level and the reproducibility at the mean of the previous chart using $\bar{x}_{\text{known}}$.

   NOTE Annex A shows a worked example including the F-test to pool the sigmas.

   If the ratio of Reproducibility_ $\bar{x}_{\text{stage1}}$ / Reproducibility_ $\bar{x}_{\text{known}}$ is between 0,85 and 1,15 then use the F-test to assess if s_{stage1} is statistically indistinguishable versus s_{known}.

   If the F-Test to pool s_{stage1} and s_{known} passes, pool both standard deviations (follow the procedure in Annex B). Assign the pooled standard deviation s_{pool} as the standard deviation s_{chart} to be used to construct the limits in step 9).

   If there is no prior information on s_{known} for this type of material, or, if the F-test fails then use s_{stage1} as s_{chart} for step 9).

9) Create the I-Chart for this batch of QC samples by assigning $\bar{x}_{\text{stage1}}$ from step 7) as $\bar{x}_{\text{chart}}$ for this step; then, overlaying the centre line represented by $\bar{x}_{\text{chart}}$, and the two control limits represented by $\bar{x}_{\text{chart}} \pm 3 \cdot s_{\text{chart}}$ onto the run chart in step 3). If the EWMA sensitivity enhancement strategy is

used, construct and plot the EWMA line with its associated control limits placed at $\bar{x}_{chart} \pm 1{,}5 \cdot s_{chart}$ onto the run chart as well.

10) Label the zones in the I-chart as follows:

 — Zone C: $\bar{x}_{chart} \pm 1 \cdot s_{chart}$ (exclusive)

 — Zone B: $\bar{x}_{chart} + 1 \cdot s_{chart}$ (inclusive) to $2 \cdot s_{chart}$ (exclusive); $\bar{x}_{chart} - 1 \cdot s_{chart}$ (inclusive) to $- 2 \cdot s_{chart}$ (exclusive).

 — Zone A: $\bar{x}_{chart} + 2 \cdot s_{chart}$ (inclusive) to $3 \cdot s_{chart}$ (exclusive); $\bar{x}_{chart} - 2 \cdot s_{chart}$ (inclusive) to $- 3 \cdot s_{chart}$ (exclusive).

11) Compute the *MR* results using all non-rejected results from Step 5).

12) Compute the average using all *MR* results and designate this as $\overline{MR}_{stage1}$.

13) If s_{pool} is used from step 8), use the weighted average $\overline{MR}_{wtd}$ computed from $\overline{MR}_{known}$ corresponding to the s_{known} and $\overline{MR}_{stage1}$ as the $\overline{MR}_{chart}$ to be used to construct the MR-chart in step 14). Otherwise, use $\overline{MR}_{stage1}$ as $\overline{MR}_{chart}$ for this batch of QC material.

14) Create the MR-chart by overlaying the lines represented by $\overline{MR}_{chart}$ and upper control limit at 3,27 $\cdot \overline{MR}_{chart}$ onto a run chart plot of all the MR values computed in step 11).

15) If all of the in-statistical-control conditions (see 4.2.4) are met, proceed to Stage 2. Otherwise, investigate and mitigate root causes for failure, then repeat from step 1).

NOTE See Annex A and B for a detailed illustration of Stage 1 steps 3) to 15) and related statistical techniques.

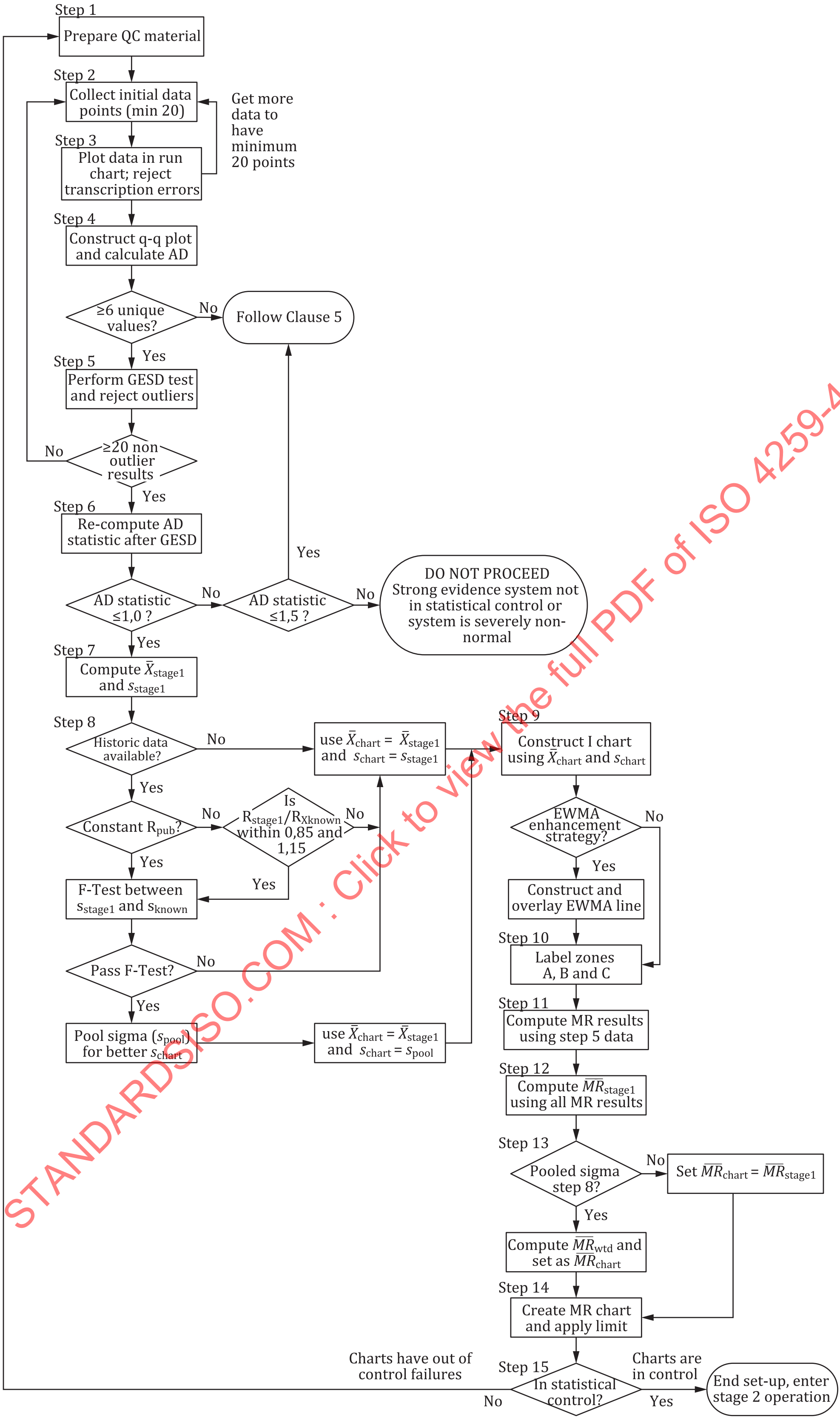


Figure 2 — Flow chart of Stage 1 of control chart work process

4.3.3 Stage 2 of control chart work process

4.3.3.1 Operation

In the operation mode, the primary objective is to detect abnormal events by:

- the immediate evaluation of a new QC sample result and its corresponding MR result against the action limits of the I-chart and MR-chart respectively;
- the immediate evaluation of whether any action is required from the chosen sensitivity enhancement Strategy (1 or 2).

The interpretation and immediate response associated with violation of the chart action limits, or, 'action required' outcome from enhancement Strategy 1 or 2, are listed below:

- Violation of I-chart limit: single result at or outside $\bar{x}_{\text{chart}} \pm 3\, s_{\text{chart}}$
  - Interpretation: a unique, sudden, event has occurred that caused the test result to deviate from the centre line by an amount that is not plausible due to common causes.
  - Immediate response: re-analyse a new QC sample to confirm unique event.
    - If violation still persist, declare the test process is out of statistical control. The test process should be taken out of service until the problem is investigated and resolved.
    - If re-analysis result is back in zones C or B as specified above [see 4.3.2 step 10)], the out of statistical control situation is not confirmed. Do not declare the process out of statistical control. However, ensure this event is duly recorded.
    - For the purpose of Stage 2 Annex B - Maintenance:
    - If the system is declared out of statistical control, exclude both the initial and re-analysis result.
    - If out of statistical control is not confirmed, exclude the initial out of control result and use only the re-analysis result if the initial out of control result exceeds the control limit by an amount > 0,25 s_{chart} and there is no MR upper control limit exceedance associate with either the original or the retest result. This is for the purpose of minimising the rejection of legitimate data. Otherwise, exclude the re-analysis result and use only the initial out of control result.

      In the case of redefining control limits, care shall be taken to ensure that initial points that exceed the control limit by ≤ 0,25 s_{chart} be included in the performance re-evaluation and the re-analysis points rejected. This approach ensures that true system performance is evaluated and not just points that were within limits leading to ever decreasing sigma values with no identification of system deterioration.
- Violation of MR-chart upper limit: single MR outside its upper action limit:
  - Interpretation: an unusually large difference has occurred between the current result and the previous result; this event is usually (but not necessarily) associated with an I-chart violation;
  - Immediate response: re-run a new QC sample if this is not associated with an I-chart violation. Investigate for possible causes of large step change:
    - If it is determined that this is not due to QC sample integrity, or, if this is not caused by an I-chart violation, due to the higher false alarm rate of this statistic, 5 or more of this event over the past 12 is reasonable statistical evidence that the variation (precision) of the process has deteriorated. Perform an on-demand precision (variance) comparison using the F-test (similar to precision comparison in maintenance mode and illustrated in Annex B) between the most recent 20 in-statistical-control results versus the current chart variance $(s_{\text{chart}})^2$.

"Action required" from I-chart sensitivity enhancement strategies (either 1 or 2)

— Interpretation: it is highly possible that the current process centre has moved away from the expected average ($\bar{x}_{chart}$) established from stage 1.

— Immediate response: confirm if this violation is instrument or QC sample related by testing CRM's or stable retains of PT or previously tested production samples that are known to have been properly stored in a manner that preserves the property of interest during storage. Compare the result obtained to the value expected. For certified reference materials (CRM) or proficiency testing (PT) samples, use the assigned reference value (ARV) as the value expected. For retain of previously tested production sample, use the previous test result.

  — If the difference between the test result obtained and the value expected is greater than $1{,}5 \cdot s_{chart}$ for CRM or PT retain, or greater than $2 \cdot s_{chart}$ for previously tested production sample, conclude that this "action required" signal is likely not due to QC sample integrity. Declare the test process is out of statistical control. The test process should be taken out of service until the problem is investigated and resolved. CRM/PT with ARV within working range of s_{chart} is preferred but not mandatory.

  — If the difference between the test result obtained versus the value expected for CRM or PT retain is $\leq 1{,}5 \cdot s_{chart}$, or $\leq 2 \cdot s_{chart}$ for previously tested production sample, conclude that the QC sample integrity is suspect. Switch to a new QC supply per 4.4 on QC material batch change.

4.3.3.2 Maintenance

4.3.3.2.1 General

In the maintenance mode, the primary objective is to reduce the uncertainties of the statistics used for I-chart parameters (centre, control limits) through periodic statistical assessment of accrued in-statistical-control data. Two scenarios are described in 4.3.3.2.2 and 4.3.3.2.3.

4.3.3.2.2 Scenario 1

A minimum of 20 new in-statistical-control QC results that are not used to compute the control chart limits has been accrued while the control chart is in Operation mode (i.e.: an *active* control chart) for the same batch of control sample.

Under this scenario, the current control chart is actively being populated by results from the same batch of QC material. Condition on the assumption that both the test process and this QC material remain unchanged, the new accrued in-statistical-control data represents a 'repeated statistical sampling' of the same target population of data. Therefore, the control chart centre and control limits may be recalculated and updated on a go-forward basis using more reliable statistics computed using a larger data set from the same target data population by combining the new accrued in control data with the data used to update the current control limits. Combining new accrued data in the aforementioned manner is condition upon no compelling evidence that invalidates the "remain unchanged" assumption, which can be supported by 'not statistically significant' outcomes from the following statistical tests at the 0,05 significance level:

First, perform the F-test of variance of new accrued in-statistical-control data versus the variance used to compute the current control limits. If F-test is not significant, pool the variances, then perform the *t*-test of the average ($\bar{x}_{new}$) of the new accrued data versus the current control chart centre ($\bar{x}_{chart}$) using the pooled standard deviation. If the *t*-test is not significant, re-compute $\bar{x}_{chart}$, s_{chart}, and $\overline{MR}_{chart}$ using all in-control data. Update the control chart limits accordingly.

If either test is significant, investigate and take corrective actions as appropriate. Re-start the control chart process afterwards. If it is determined that the root cause for the significant outcome for either or both tests is due to control sample integrity (changed or degraded over time), follow 4.4. Otherwise, restart the control chart process from step 5) in 4.3.2 using the new accrued data.

NOTE Based on statistical theory, if the testing process remains in-statistical-control, the control chart remains in control. However, the reverse isn't always true. That is because statistics from which action limits for control charts are calculated do not have sufficient power to detect small magnitude of sustained mean shift or precision change. Re-computation and update of the current active control chart limits is justified if both F and *t*-test with additional data are not significant.

Follow the procedure in Annex B on how to perform appropriate F and *t* tests.

4.3.3.2.3 Scenario 2

When it is necessary to archive the current control chart, this can be done by carrying out the following activities that require s_{known}, df_s_{known} and $\overline{MR}_{\text{known}}$ to be well defined from > 50 df, so that a revised value for s_{known} can be calculated:

1) Assign the current $\bar{x}_{\text{chart}}$, s_{chart} $\overline{MR}_{\text{chart}}$, total number of retained chart results (n) as final achieved chart average $\bar{x}_{\text{achieved}}$, standard deviation s_{achieved}, and $\overline{MR}_{\text{achieved}}$, n_{achieved}, and $df\_s_{\text{achieved}} = (n_{\text{achieved}} - 1)$ for the specific batch of QC material before archiving the control chart.

2) If s_{chart} for this archived control chart when it is initially deployed [see 4.3.2, step 8)] is obtained by pooling with an existing s_{known}, continue with the following actions in 2 a) and b). Otherwise go to step 3).

   a) Update the current s_{known}, $\overline{MR}_{\text{known}}$ by replacing them with s_{achieved}, $\overline{MR}_{\text{achieved}}$;

   b) increase the df_s_{known} by adding df_s_{achieved} to the previously existing df_s_{known}.

3) If s_{chart} for this archived control chart when it is initially deployed [see 4.3.2, step 8)] is not obtained by pooling with an existing s_{known}, assign s_{achieved} as the s_{known} for the QC material associated with the archived control chart. Assign $\bar{x}_{\text{achieved}}$ and df_s_{achieved} to the working range and df for this s_{known}.

4.4 QC material batch change transition

4.4.1 General

Since control limit calculations for the I-chart require a centre value established by a minimum of 20 in-statistical-control results, and, a new batch of QC material with exactly the same property as the current batch is generally not available, especially if the material is taken from different manufacturing production runs, a special transition procedure is necessary to ensure control chart coverage of the testing process remain uninterrupted while the centre value for a new batch of QC material is being established.

This subclause prescribes three procedures. Anyone of these procedures may be used to address the transition to a new QC batch.

Procedures 2 and 3 become active upon arrival of the second result. The first result shall be independently validated by either a single overlap from the previous control chart per Procedure 1, or, a CRM test result that is within the reference value ± 1,5 s where s is the current s_{known}. The CRM shall have a reference value that is within the working range of the s_{known}.

NOTE See Annex A for details on Procedures 2 and 3.

4.4.2 Procedure 1, concurrent testing

4.4.2.1 Collect and prepare a new batch of QC samples. While this step may be performed at any time, it shall be completed when the current QC material supply remaining can support no more than 20 analyses.

4.4.2.2 Each time a current QC sample is tested, if the result (current QC sample) is in-statistical-control, immediately test and record a result for the new material. The result for the new material is validated as fit-for-use based on the result from the current QC sample is in-statistical-control.

4.4.2.3 After a minimum of 20 validated results for the new batch in 4.4.2.2 becomes available, complete steps 7) to 15) in 4.3.2.

4.4.3 Procedure 2, Q-chart

The Q-chart described uses Case II of Reference [6] where μ is unknown and $\sigma = \sigma_o$.

Case II is exactly the situation for transitioning to a new QC material with s_{known} established over QC testing history of similar material (Stage 2 - control chart maintenance), but the $\bar{x}_{\text{new}}$ is unknown.

To use the Q-chart for transition, s_{known} with an associated df_s_{known} of at least 70 should be used as σ_o in the Q-statistic described in Reference [6].

The Q_r statistic, calculated as each new result arrives, is the standard normal deviate variable Z, with $\mu = 0$ and $\sigma = 1$.

The Q_r statistic has no associated measurement units and can be plotted on a standardised control chart with fixed, forward looking control limits computed using $\bar{x} = 0$ and $s = 1$.

After a minimum of 20 in-control Q_r, complete steps 7) to 15) in 4.3.2.

4.4.4 Procedure 3, dynamically updated I-chart with EWMA

This is essentially an I-chart and EWMA (Strategy 2) with varying control chart limits that are updated with the arrival of each new result. The in-statistical-status of the new result is judged using limits computed from all previous results.

If the new result is in control, the control limits are updated by incorporating the new result into the calculation for the control limits, to be used to judge the next result. This dynamic update is to account for the change in the standard error associated with the centre line ($\bar{x}$), which is steadily decreasing as the number of results used for its computation increases. s_{known} used to position the control limits does not change, it is only the numerical value for $\bar{x}$ and the contribution of its associated standard error towards the position of the control limits that are changing.

The system is declared in control if the following conditions are met: 1) the new QC result is inside the control limits computed using all previous results; 2) new result and all previous results are inside the updated control limits after 1) is met.

If any result and/or EWMA are outside its respective limits, the current system status is declared to be out of statistical control. While this approach can facilitate plotting the results in its actual measurement units, the chart interpretation is non-intuitive and can be confusing.

For this procedure, the control limits for the EWMA is invoked beginning with the 6th in control x.

5 Guidance for insufficient variation or non-normal data

5.1 General requirement

The control charts described in 4.2 require data with sufficient resolution to permit variation to be observable in a statistically meaningful manner, and, (data) can be reasonably represented by the Normal distribution.

Statistically meaningful variation implies that the total number of unique values in the data set should be sufficiently large (≥6).

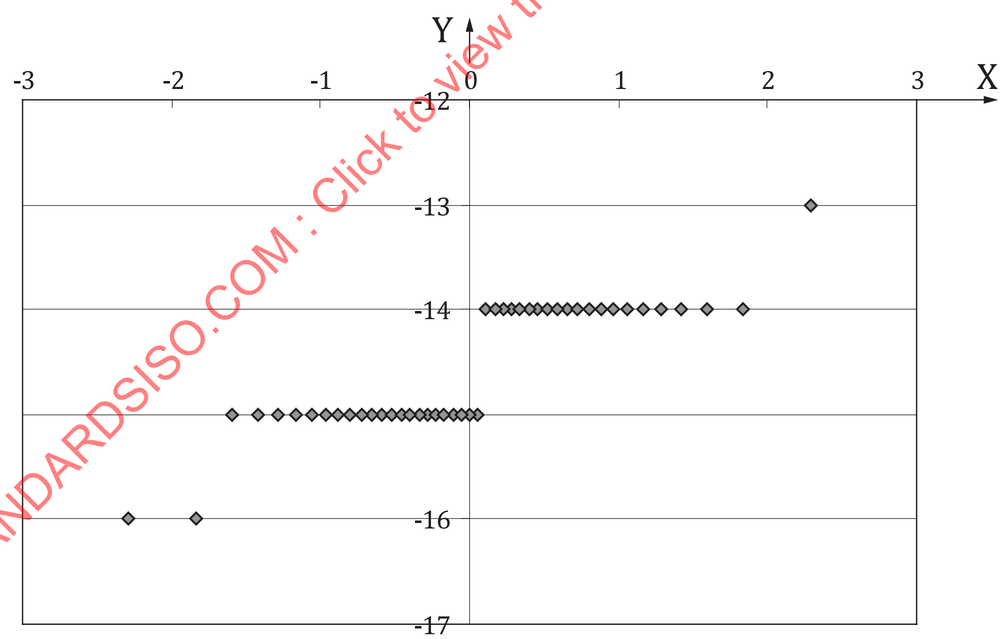
Data set with < 6 unique values are considered to be data with insufficient resolution by this document.

Data set with *AD* > 1,0 are considered to be non-normal data by this document.

5.2 How to deal with insufficient variation or non-normal data

5.2.1 Insufficient variation

For test methods with rounding instructions, the requirement in 5.1 (more than six unique values) may not be met, which means the data lacks sufficient resolution to provide statistically meaningful variation for the application of control charts as described in 4.2. Some examples are cloud, pour point, and digital density data. Manual cloud results, for example, are rounded to the nearest whole number (i.e. no decimals). The insufficient resolution problem is visually obvious by looking at the q-q plot as it will have a few horizontal lines or clusters of data that are numerically identical. See Figure 3 for an example.



Key

X Z score of results sorted smallest to largest

Y numerical value of results corresponding to each z-score

Figure 3 — Example of data clusters

For the purpose of applying the control charts in 4.2, the first and preferred approach is to increase data resolution by obtaining an additional decimal in the results. It should be noted that because results are used for internal QA control charting purposes, this should not be considered as a deviation from test method reporting requirements since these results are not reported to external customers.

If obtaining an additional decimal is not possible, or, does not provide sufficient unique values in the data set, this document recommends using the minimum and maximum value in the data set as action limits to be placed on the run chart in step 3).

IMPORTANT — Using the minimum and maximum is a special case of using the 1st and 99th p-tile in data set to set action limits since for a small data set (<100), these p-tiles default to the minimum and maximum value in the data set. There is no safeguard against outliers using this approach. Therefore, judgement is required to ensure these limits are reasonable; however, approaches such as median absolute deviation (MAD) may be used to identify potential outlier values.

A run chart with action limits as described above is not considered to be a control chart as specified by this document and should not be labelled as such.

5.2.2 Non-normal data

For the situation where the total number of unique values in the data set is ≥ 6, and, 1,0 < AD < 1,5, a statistical professional that is familiar with the test method metrology principle and the business application requirement for the test method should be consulted.

If the run chart in 4.3.2 does not show any discernible trend (upwards or downwards) over time, or, does not show a distinct break from one level to another at a specific location in the chronological sequence, the minimum and maximum value approach described above may be useful in some cases.

Per 4.3.2 step 6), this document does not address data set with ≥ 6 unique values and AD > 1,5, as this is strong statistical evidence that either the system is not in-statistical-control, or, the process results distribution is severely non-normal.

Annex A
(informative)

Details of the control chart work process

A.1 Stage 1 (see 4.3.2)

This annex details and gives examples for steps 3) to 15) of the Stage 1 as presented in 4.3.2.

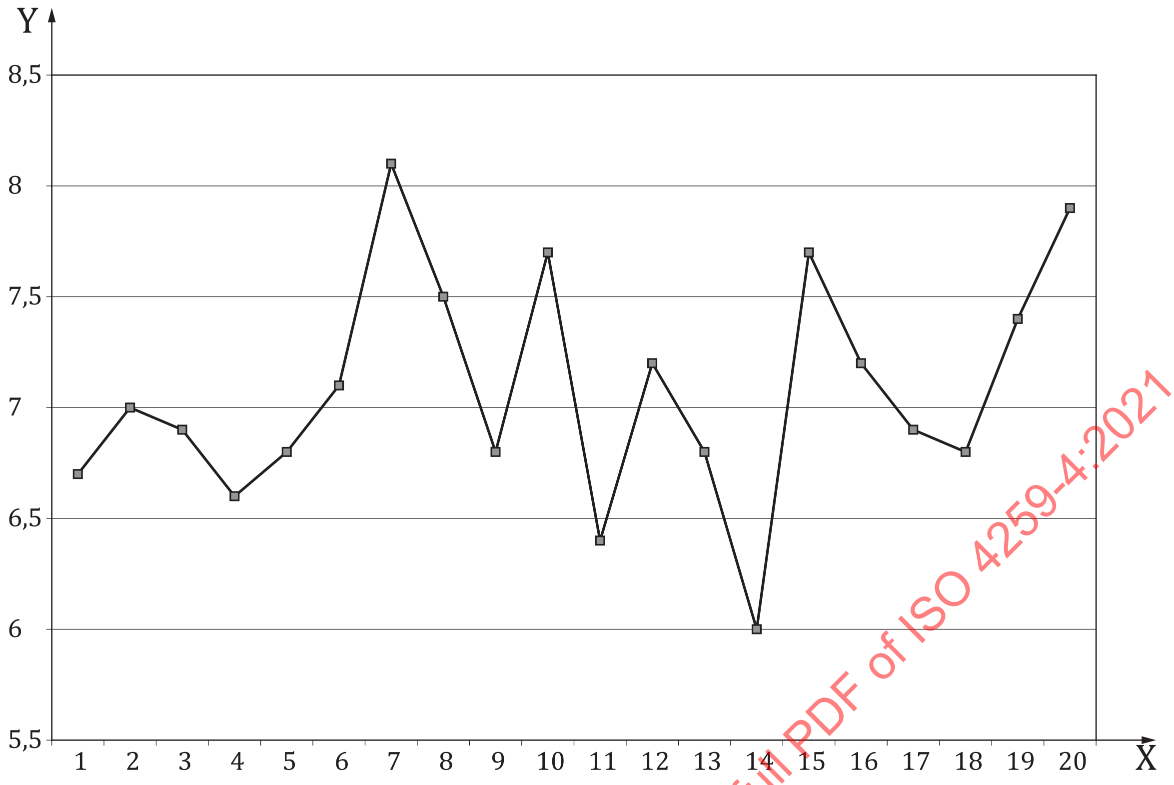
Step 3) reads: "Plot the individual results chronologically (this is called a run chart) and study the plot for any visually discernible transcription errors. Obvious transcription errors should be corrected or discarded". The data used for the example are given in Table A.1 and plotted in Figure A.1.

Table A.1 — Run chart data

i	*X* mg/kg
1	6,7
2	7,0
3	6,9
4	6,6
5	6,8
6	7,1
7	8,1
8	7,5
9	6,8
10	7,7
11	6,4
12	7,2
13	6,8
14	6,0
15	7,7
16	7,2
17	6,9
18	6,8
19	7,4
20	7,9

i is the chronologically ordered number for the observation.

X is a single result from testing a QC sample.



Key

X number of observations in sequential order

Y numerical value of observation results

Figure A.1 — Run chart from Table A.1 results of first 20 observations

Step 4) reads "Construct a quantile-quantile (q-q) plot and compute the Anderson-Darling (AD) statistic". Outlined below is the procedure for creating a q-q plot using the data from the run chart as in Table A.1 (see also Reference [7]). See step 6) for AD computation procedure.

4a) Order the data from smallest to largest (n = total number of observations = 20).

4b) Create an index i next to the ordered data where i will take on values from 1 through 20, with the lowest value assigned i = 1 and the highest assigned i = 20.

4c) Calculate $f_i = (i - 0{,}5)/n$ for each observation.

4d) Obtain from the cumulative distribution version of a standard normal distribution table (μ = 0, σ = 1) the value of z_i for each f_i. An easier approach is to use the spreadsheet function NORMSINV function to compute the z_i values as shown in Table A.2.

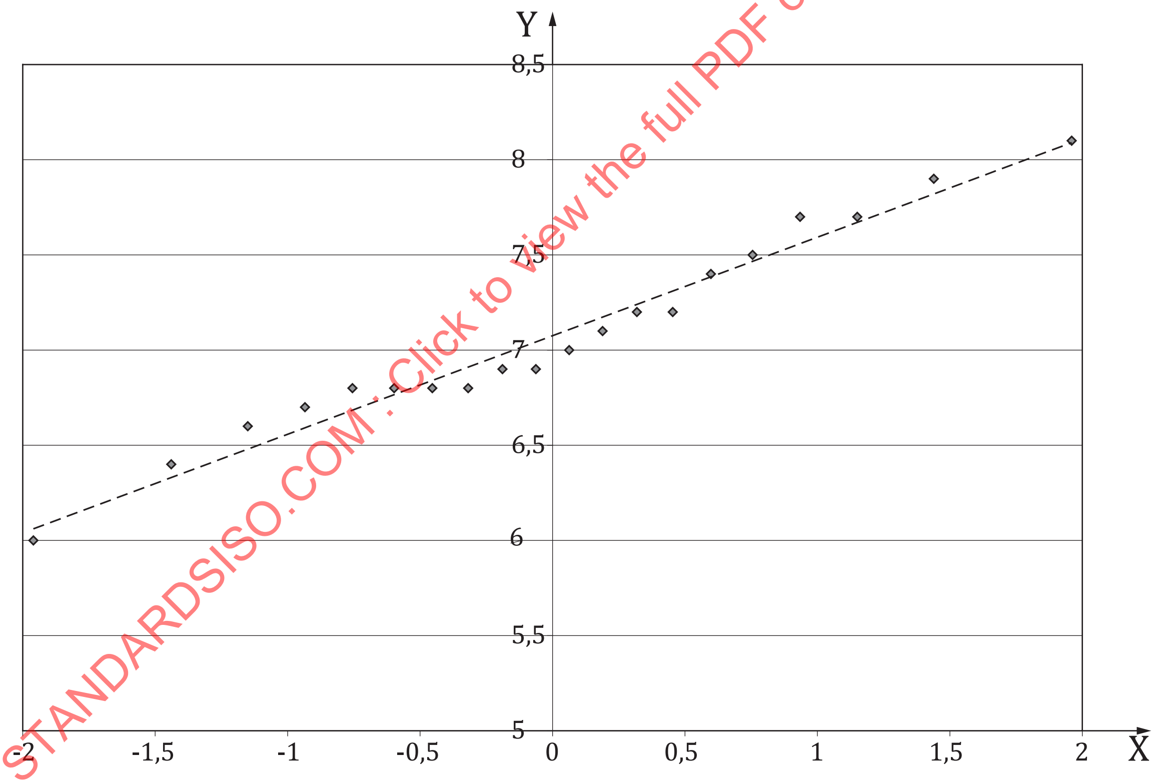
4e) Plot each ordered data value on the y-axis against its z_i value obtained in 4d). on the x-axis using ordinary linear graph paper or using computer programs. This creates the q-q plot (see Figure A.2).

Table A.2 — Computing of the *z*-values

Original data	Ordered data	Index *i*	$f_i = (i - 0{,}5)/n$	z_i = NORMSINV(f_i)
6,7	6,0	1	0,025	−1,960
7,0	6,4	2	0,075	−1,440
6,9	6,6	3	0,125	−1,150
6,6	6,7	4	0,175	−0,935
6,8	6,8	5	0,225	−0,755
7,1	6,8	6	0,275	−0,598

Table A.2 *(continued)*

Original data	Ordered data	Index *i*	$f_i = (i - 0,5)/n$	z_i = NORMSINV(f_i)
8,1	6,8	7	0,325	−0,454
7,5	6,8	8	0,375	−0,319
6,8	6,9	9	0,425	−0,189
7,7	6,9	10	0,475	−0,063
6,4	7,0	11	0,525	0,063
7,2	7,1	12	0,575	0,189
6,8	7,2	13	0,625	0,319
6,0	7,2	14	0,675	0,454
7,7	7,4	15	0,725	0,598
7,2	7,5	16	0,775	0,755
6,9	7,7	17	0,825	0,935
6,8	7,7	18	0,875	1,150
7,4	7,9	19	0,925	1,440
7,9	8,1	20	0,975	1,960



Key

X Z score of results sorted smallest to largest

Y numerical value of results corresponding to each z-score

Figure A.2 — q-q plot of run chart data

Step 5) "Perform a formal statistical assessment for outliers using GESD technique for outliers according to ISO 4259-1. The recommended maximum number of outliers for 20 to 25 observations is 3 at 0,01 significance."

The procedure for GESD assessment of data from the run chart above is described below and illustrated in Table A.3. In addition to 4.3.2, see Reference [8]:

5a) Set the maximum number of outliers r = 3.

5b) Set current cycle index i = 1.

5c) Calculate the quantity $T = |\text{observation} - \bar{x}| / s$ for every member of the data set in the current cycle, where: $\bar{x}$ and s are average and standard deviation computed using all members in the data set in the current cycle respectively

5d) Identify the observation with the largest T. Designate this as $T_{i\ \text{max}}$ (i.e.: maximum T for the i^{th} cycle.)

5e) Remove the observation identified in 5d) from the data set.

5f) Increase current cycle index i by 1: i.e. $i = i + 1$

5g) Repeat steps 5c) to 5f) using remaining data up to and including $i = r = 3$

5h) Upon completion of 5g), beginning with $T_{3\ \text{max}}$, maximum T value in last cycle, and working backwards ($T_{2\ \text{max}}$, $T_{1\ \text{max}}$), compare this maximum value versus the critical value for the specific cycle (λ_i) listed in Table A.4 corresponding to the size of the data set. For this example, the data set size is 20.

5i) Identify the highest cycle for which $T_{i\ \text{max}}$ exceeds its limit value. The observation associated with the $T_{i\ \text{max}}$ for that cycle and all observations associated with the $T_{i\ \text{max}}$'s for all previous cycles up to and including cycle 1 are considered to be outliers.

Table A.3 — Illustration of GESD assessment

average for cycle =	7,08		7,13		7,08	
std dev for cycle =	0,52		0,47		0,42	
ordered data from Table A.1	**cycle 1**	***T***	**cycle 2**	***T***	**cycle 3**	***T***
6,0	6,0	2,06 = $T_{1\ \text{max}}$				
6,4	6,4	1,29	6,4	1,56	6,4	1,62
6,6	6,6	0,91	6,6	1,13	6,6	1,14
6,7	6,7	0,72	6,7	0,92	6,7	0,90
6,8	6,8	0,53	6,8	0,71	6,8	0,66
6,8	6,8	0,53	6,8	0,71	6,8	0,66
6,8	6,8	0,53	6,8	0,71	6,8	0,66
6,8	6,8	0,53	6,8	0,71	6,8	0,66
6,9	6,9	0,34	6,9	0,49	6,9	0,43
6,9	6,9	0,34	6,9	0,49	6,9	0,43
7,0	7,0	0,14	7,0	0,28	7,0	0,19
7,1	7,1	0,05	7,1	0,07	7,1	0,05
7,2	7,2	0,24	7,2	0,15	7,2	0,29
7,2	7,2	0,24	7,2	0,15	7,2	0,29
7,4	7,4	0,62	7,4	0,57	7,4	0,77
7,5	7,5	0,81	7,5	0,79	7,5	1,01
7,7	7,7	1,20	7,7	1,21	7,7	1,49
7,7	7,7	1,20	7,7	1,21	7,7	1,49

Table A.3 *(continued)*

7,9	7,9	1,58	7,9	1,64	7,9	1,97 = $T_{3\ max}$
8,1	8,1	1,96	8,1	2,06 = $T_{2\ max}$		
λ_i		3,00		2,97		2,93

Beginning with $T_{3;max}$, it is compared to its corresponding critical value λ_3 of 2,93. Since $T_{3\ max}$ is less than its critical value selected from Table A.4, $T_{2\ max}$ is compared with its critical value of 2,97, which is also less than its critical value and therefore $T_{1\ max}$ is checked. Since no λ_i is exceeded, it can be concluded there are no outliers in this data set.

Table A.4 — Critical λ_i values for n_0

cycle = >	1	2	3	4
n_0	Critical λ_i	Critical λ_i	Critical λ_i	Critical λ_i
20	3,00	2,97	2,93	2,89
21	3,03	3,00	2,97	2,93
22	3,06	3,03	3,00	2,97
23	3,09	3,06	3,03	3,00
24	3,11	3,09	3,06	3,03
25	3,14	3,11	3,09	3,06
n_0 is the number of results prior to commencement of GESD evaluation.				

Step 6) reads “Confirm the goodness-of-fit of the Normal distribution using the Anderson-Darling (AD) statistic computed from at least 20 non-outlier results.”

The AD statistic is represented by the symbol A^{2*}, computed using the following:

$$A^{2*} = A^2\left(1+\frac{0,75}{n}+\frac{2,25}{n^2}\right) \quad \text{(A.1)}$$

where:

$$A^2 = \frac{-\left\{\sum_{i=1}^{n}(2i-1)\left[\ln(p_i)+\ln(1-p_{n+1-i})\right]\right\}}{n} - n$$

p_i is the cumulative probability of the standard normal deviate z_i

$$z_i = \frac{(x_i-\bar{x})}{s} \quad \text{(A.2)}$$

where

$\bar{x}$ is the average number of observations;

s is the standard deviation of observations.

The AD calculation for the 20 observations from the run chart is illustrated in Table A.5.

where: $H = p_{n+1-i}$

$$V = (2i-1)[\ln(p_i) + \ln(1 - p_{n+1-i})] \tag{A.3}$$

The data in

$A^2 = 0{,}328$ $A^{2*} = 0{,}342$

Table A.5 — Observations during the chart

$\bar{x}$ = 7,075

s = 0,522

n = 20

Original Data	Ordered Data	Index i	z_i	p_i	ln(p_i)	(n+1-i)	H	(1 - H)	ln(1 - H)	(2i-1)	V
6,7	6,0	1	−2,059	0,020	−3,925 5	20	0,975	0,025	−3,697	1	−7,623
7,0	6,4	2	−1,293	0,098	−2,322 8	19	0,943	0,057	−2,865	3	−15,562
6,9	6,6	3	−0,910	0,181	−1,706 9	18	0,884	0,116	−2,158	5	−19,323
6,6	6,7	4	−0,718	0,236	−1,442 8	17	0,884	0,116	−2,158	7	−25,203
6,8	6,8	5	−0,527	0,299	−1,206 8	16	0,792	0,208	−1,571	9	−25,002
7,1	6,8	6	−0,527	0,299	−1,206 8	15	0,733	0,267	−1,321	11	−27,809
8,1	6,8	7	−0,527	0,299	−1,206 8	14	0,595	0,405	−0,903	13	−27,426
7,5	6,8	8	−0,527	0,299	−1,206 8	13	0,595	0,405	−0,903	15	−31,646
6,8	6,9	9	−0,335	0,369	−0,997 7	12	0,519	0,481	−0,732	17	−29,407
7,7	6,9	10	−0,335	0,369	−0,997 7	11	0,443	0,557	−0,585	19	−30,071
6,4	7,0	11	−0,144	0,443	−0,814 5	10	0,369	0,631	−0,460	21	−26,764
7,2	7,1	12	0,048	0,519	−0,655 7	9	0,369	0,631	−0,460	23	−25,660
6,8	7,2	13	0,239	0,595	−0,519 8	8	0,299	0,701	−0,355	25	−21,883
6,0	7,2	14	0,239	0,595	−0,519 8	7	0,299	0,701	−0,355	27	−23,633
7,7	7,4	15	0,623	0,733	−0,310 3	6	0,299	0,701	−0,355	29	−19,308
7,2	7,5	16	0,814	0,792	−0,232 9	5	0,299	0,701	−0,355	31	−18,240
6,9	7,7	17	1,197	0,884	−0,122 8	4	0,236	0,764	−0,270	33	−12,948
6,8	7,7	18	1,197	0,884	−0,122 8	3	0,181	0,819	−0,200	35	−11,306
7,4	7,9	19	1,580	0,943	−0,058 7	2	0,098	0,902	−0,103	37	−5,988
7,9	8,1	20	1,964	0,975	−0,025 1	1	0,020	0,980	−0,020	39	−1,756
										-ΣV/n	20,328

Step 7) reads: “Compute the average ($\bar{x}_{\text{stage1}}$) and standard deviation (s_{stage1}) statistics using the non-rejected results from 5) for the new batch of QC material”.

$\bar{x}_{\text{stage1}}$ = 7,075 computed from run chart observations

s_{stage1} = 0,522 computed from run chart observations

$df\_s_{\text{stage1}} = n_{\text{stage1}} - 1 = 20 - 1 = 19$

Step 8) is as follows: “If $\bar{x}_{\text{stage1}}$ is within the working range (see 4.3.3.2, Scenario 2) associated with a known standard deviation (s_{known}) for this type of QC material, ...”

The s_{known} for this type of QC material accrued from previous control charts is:

s_{known} = 0,623; working range: x̄ from 7,132 to 7,305; $\overline{MR}_{known}$ = 0,487 df_{known} = 75

1,5(s_{known}) = 0,935

The total range spanned by $\bar{x}_{stage1}$ + the working range for s_{known} is (7,305 – 7,075) = 0,23 < 1,5(s_{known}), proceed to determine if s_{stage1} is statistically indistinguishable from s_{known}, following the F-test procedure in Annex B.

$F = (0{,}623/0{,}522)^2 = 1{,}424$

$F_{cri.}$ = 2,24

Since 1,424 is less than 2,30, conclude s_{stage1} is statistically indistinguishable from s_{known}. Since it is desired to use a more reliable s, proceed to compute s_{pool} by following the technique in Annex B as follows:

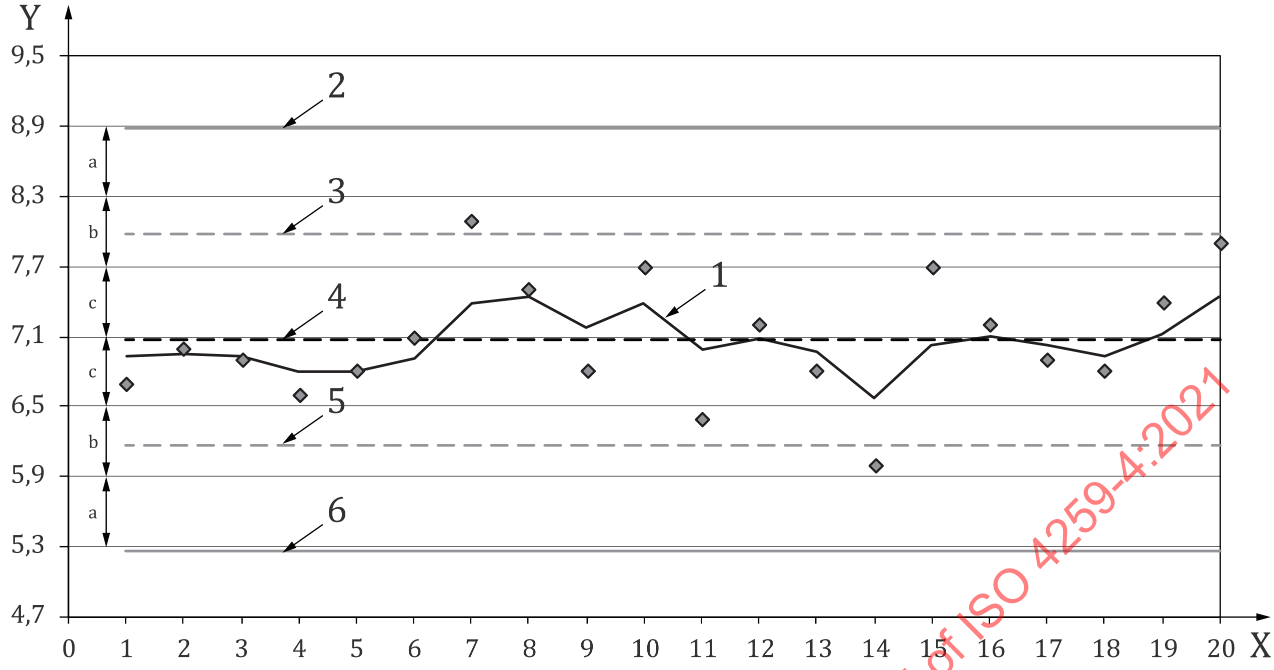
$$\text{pooled variance} = \frac{(df\_s_{known})(s_{known})^2 + (df\_s_{stage1})(s_{stage1})^2}{(df\_s_{known} + df\_s_{stage1})} \tag{A.4}$$

$= (75*(0{,}623)^2 + 19*(0{,}522)^2)/\ (75 + 19)$

$s_{pool} = \sqrt{pooled\ variance}$ = 0,604 and this pooled sigma shall be used as s_{chart}.

$df\_s_{pool} = df\_s_{known} + df\_s_{stage1} = 75 + 19 = 94$

Step 9): "Create the I-Chart for this batch of QC by assigning $\bar{x}_{stage1}$ from step 7) above as $\bar{x}_{chart}$ for this step; then, overlaying the centre line represented by $\bar{x}_{chart}$, and the two action limits represented by $\bar{x}_{chart} \pm 3 \cdot s_{chart}$ onto the run chart in step 3) above. If the EWMA sensitivity enhancement strategy is used, construct and plot the EWMA line with its associated action limits placed at $\bar{x}_{chart} \pm 1{,}5 \cdot s_{chart}$ and overlay them onto the run chart as well. See Figure A.3.



Key

X	observations	3	UCL_EWMA (value at 7,98)
Y	sequence of testing	4	mean ($\bar{x}$)
◆	test result (X)	5	LCL_EWMA (value at 6,17)
1	EWMA	6	LCL_X (value at 5,26)
2	UCL_X (value at 8,89)		
a	Zone A.		
b	Zone B.		
c	Zone C.		

Figure A.3 — I chart generated ($\bar{x}$ = 7,075, s = 0,604)

Step 10): Label the zones in the I-chart as follows:

Zone C: $\bar{x}_{\text{chart}} \pm 1\ s_{\text{chart}}$ (exclusive)

Zone B: $\bar{x}_{\text{chart}} + 1\ s_{\text{chart}}$ (inclusive) to 2 s_{chart} (exclusive); $\bar{x}_{\text{chart}} - 1\ s_{\text{chart}}$ (inclusive) to − 2 s_{chart} (exclusive)

Zone A: $\bar{x}_{\text{chart}} + 2\ s_{\text{chart}}$ (inclusive) to 3 s_{chart} (exclusive); $\bar{x}_{\text{chart}} - 2\ s_{\text{chart}}$ (inclusive) to − 3 s_{chart} (exclusive)
"

The values for construction of the I-chart parameters are: $\bar{x}_{\text{chart}}$ = 7,075 s_{chart} = 0,604

EWMA is computed as follows: $EWMA_r = \lambda X_r + (1 - \lambda)EWMA_{r-1}$

where: λ = 0,4; X_r = the r^{th} observation, $EWMA_r$ = EWMA updated using r^{th} observation X and $EWMA_{r-1}$; $EWMA_0 = \bar{x}_{\text{chart}}$

Step 11): "Compute the MR results using all non-rejected results from step 5).

Step 12): "Compute the average using all MR results and designate this as $\overline{MR}_{\text{stage1}}$

Step 13): "If s_{pool} is used from step 8), use the weighted average $\overline{MR}_{\text{wtd}}$ computed from $\overline{MR}_{\text{known}}$ corresponding to the s_{known} and $\overline{MR}_{\text{stage1}}$ as the $\overline{MR}_{\text{chart}}$ to be used to construct the MR-chart in step 14). Otherwise, use $\overline{MR}_{\text{stage1}}$ as $\overline{MR}_{\text{chart}}$ for this batch of QC material.".

MR is computed as follows: MR_r = | X_r − X_{r-1} |; X_r is the MR value computed upon arrival of the r_{th} observation; there is no MR_1

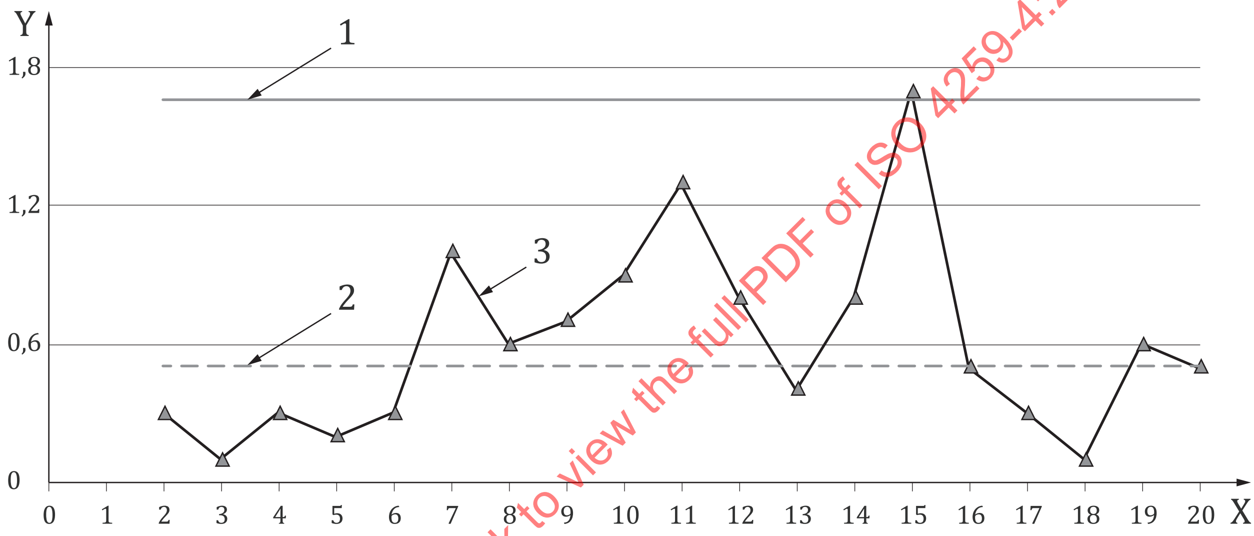
The $\overline{MR}_{stage1}$ as computed from the run chart data is 0,60

The $\overline{MR}_{known}$ associated with s_{known} is 0,487.

Since s_{pool} is used from step 8), proceed to compute $\overline{MR}_{wtd}$:

$$\overline{MR}_{wtd} = \frac{(df\_s_{known})(\overline{MR}_{known}) + (df\_s_{stage1})(\overline{MR}_{stage1})}{(df\_s_{known} + df\_s_{stage1})} = 0{,}51$$

Step 14): "Create the MR-Chart by overlaying the lines represented by $\overline{MR}_{chart}$ and upper control limit at 3,27 × $\overline{MR}_{chart}$ onto a run chart plot of all the MR values computed in step 11)". See Figure A.4.



Key

X	observations	upper line	1	$UCL\_\overline{MR} = 3{,}27\,\overline{MR}$
Y	MR	dashed line	2	$\overline{MR} = 0{,}51$
		triangled-line	3	MR results

Figure A.4 — MR chart generated

Table A.6 — Data for Figures A.3 and A.4

Obs. No. (r)	Test result (X)	EWMA	$\bar{x}$	UCL X	LCL X	UCL EWMA	LCL EWMA	MR	$\overline{MR}$	UCL MR
1	6,7	6,93	7,08	8,89	5,26	7,98	6,17			
2	7,0	6,96	7,08	8,89	5,26	7,98	6,17	0,3	0,51	1,67
3	6,9	6,93	7,08	8,89	5,26	7,98	6,17	0,1	0,51	1,67
4	6,6	6,80	7,08	8,89	5,26	7,98	6,17	0,3	0,51	1,67
5	6,8	6,80	7,08	8,89	5,26	7,98	6,17	0,2	0,51	1,67
6	7,1	6,92	7,08	8,89	5,26	7,98	6,17	0,3	0,51	1,67
7	8,1	7,39	7,08	8,89	5,26	7,98	6,17	1,0	0,51	1,67
8	7,5	7,44	7,08	8,89	5,26	7,98	6,17	0,6	0,51	1,67
9	6,8	7,18	7,08	8,89	5,26	7,98	6,17	0,7	0,51	1,67
10	7,7	7,39	7,08	8,89	5,26	7,98	6,17	0,9	0,51	1,67

Table A.6 *(continued)*

Obs. No. (r)	Test result (X)	EWMA	$\bar{x}$	UCL X	LCL X	UCL EWMA	LCL EWMA	MR	$\overline{MR}$	UCL MR
11	6,4	6,99	7,08	8,89	5,26	7,98	6,17	1,3	0,51	1,67
12	7,2	7,08	7,08	8,89	5,26	7,98	6,17	0,8	0,51	1,67
13	6,8	6,97	7,08	8,89	5,26	7,98	6,17	0,4	0,51	1,67
14	6,0	6,58	7,08	8,89	5,26	7,98	6,17	0,8	0,51	1,67
15	7,7	7,03	7,08	8,89	5,26	7,98	6,17	1,7	0,51	1,67
16	7,2	7,10	7,08	8,89	5,26	7,98	6,17	0,5	0,51	1,67
17	6,9	7,02	7,08	8,89	5,26	7,98	6,17	0,3	0,51	1,67
18	6,8	6,93	7,08	8,89	5,26	7,98	6,17	0,1	0,51	1,67
19	7,4	7,12	7,08	8,89	5,26	7,98	6,17	0,6	0,51	1,67
20	7,9	7,43	7,08	8,89	5,26	7,98	6,17	0,5	0,51	1,67

Step 15): "If all of the in-statistical-control conditions (see 4.2.4) are met, proceed to Stage 2."

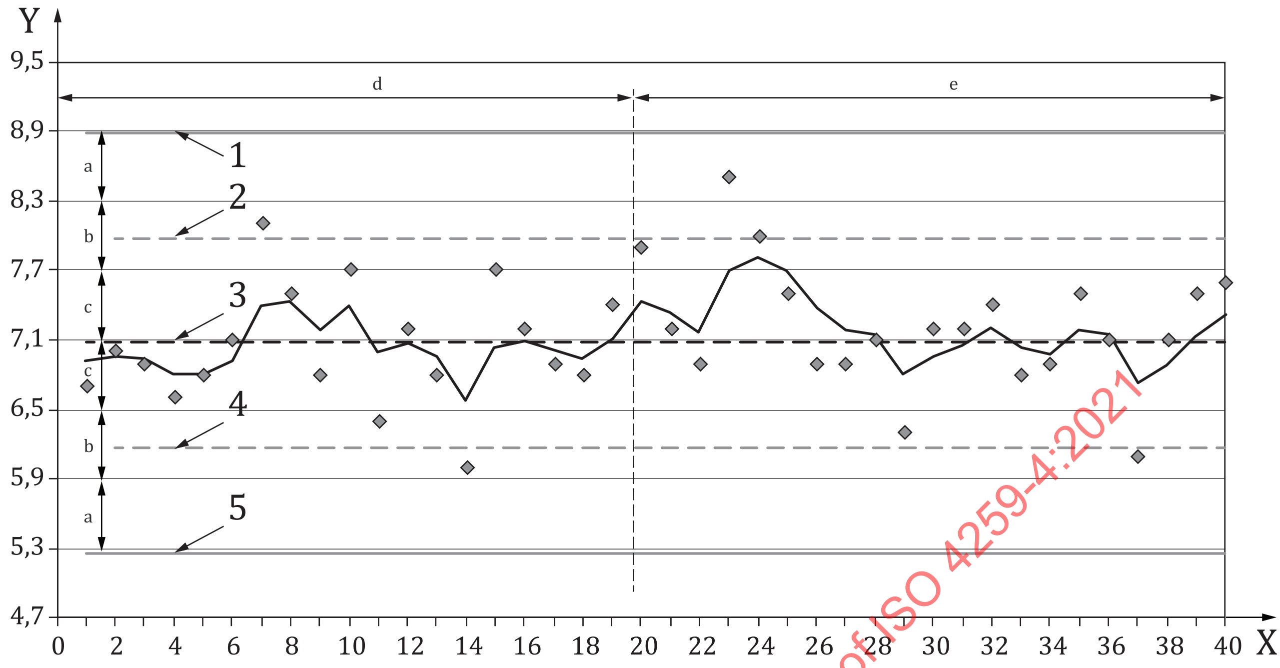
Since all conditions in 4.2.4 are met, these charts are deployed for Stage 2. Note that the single MR result exceeding (slightly) the chart control limit does not constitute an out-of-statistical-control situation.

A.2 Maintenance (see 4.3.3.2)

A.2.1 Scenario 1 (see 4.3.3.2.2)

Combining new accrued in-statistical-control QC results while the control chart is in Operation mode (i.e.: an *active* control chart) for the same batch of control sample.

See Figure A.5 for how the second stage would then look. Where in the first stage $\bar{x}$ = 7,075, s = 0,604 and df_s = 94, the new data are now: $\bar{x}$ = 7,185, s = 0,531 and df_s = 19.



Key

X	observations	2	UCL_EWMA (value at 7,98)
Y	sequence of testing	3	mean ($\bar{x}$)
◆	test result (*X*)	4	LCL_EWMA (value at 6,17)
1	UCL_*X* (value at 8,89)	5	LCL_*X* (value at 5,26)
a	Zone A.		
b	Zone B.		
c	Zone C.		
d	Stage 1 - setup data.		
e	Stage 2 - new data.		

Figure A.5 — I chart stage 2 generated

F-test to validate "*remain unchanged*" assumption for *s*:

$F = [0,6 / 0,53]^2 = 1,28$; $F_{\text{crit.}(0,05,\ 94,\ 19)} = 2,22$; not significant.

Although the approximate F-test is conducted at the 95 % (0,05) probability level (two sided), the critical F values come from the 97,5 % (0,025) percentiles of the F-statistic.

$s_{\text{pool}} = 0,592$ (see Annex B for detailed methodology on pooling).

t-test to validate "*remain unchanged*" assumption for $\bar{x}$

$t = |7,19 - 7,08| / 0,093 = 1,18$; $t_{\text{cri}\ (.025,\ 38)} = 2,02$; not significant

Re-compute control chart parameters using all accrued data by repeating steps 7 to 14 in 4.3.2 as follows:

$\bar{x}$ = average of all data = 7,13

s_{chart} from conclusion of Stage 1 = 0,604; $df\_s_{\text{chart}} = 94$

$s_{\text{new data}} = 0,531$; $df_{\text{new data}} = 19$

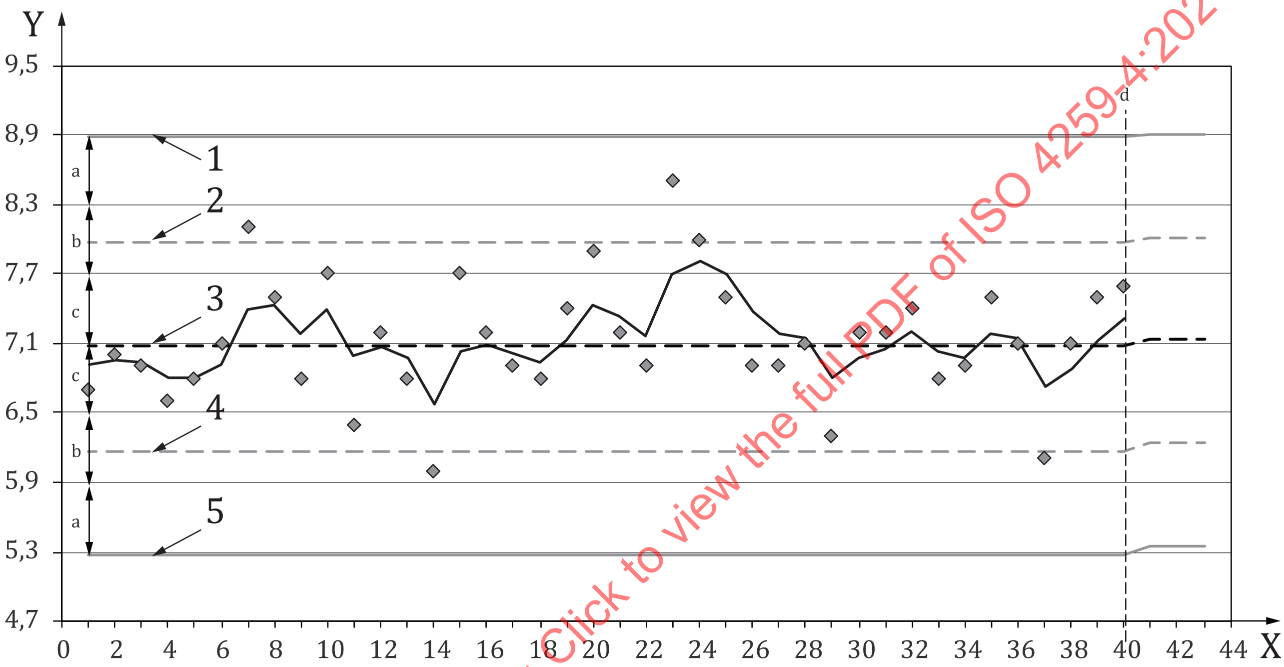
updated s = 0,592, computed from pooling of $s_{\text{new data}}$ with s_{chart} from conclusion of Stage 1

$\overline{MR}_{\text{newdata}}$ = 0,53

$\overline{MR}_{\text{chart}}$ from conclusion of Stage 1 is 0,51

updated $\overline{MR}_{\text{chart}}$ = {(19)(0,53) + (94)(0,51)} / (113) = 0,51 (rounded to 0,01).

See Figures A.6 and A.7 for the newly generated charts. In Figure A.6, the updated limits recomputed from all in-control data are given by the arrows. In Figure A.7, the lines indicate the new computed *UCL_MR* (1) and $\overline{MR}$ (2). Data for Figure A.6 and A.7 (3) are given in Table A.7.



Key

X	observations	2	UCL_EWMA (value at 7,98 to 8,02)
Y	sequence of testing	3	mean ($\bar{x}$)
◆	test result (*X*)	4	LCL_EWMA (value at 6,17 to 6,24)
1	UCL_*X* (value at 8,89 to 8,91)	5	LCL_*X* (value at 5,26 to 5,35)
a	Zone A.		
b	Zone B.		
c	Zone C.		
d	Revised limit applies to chart.		

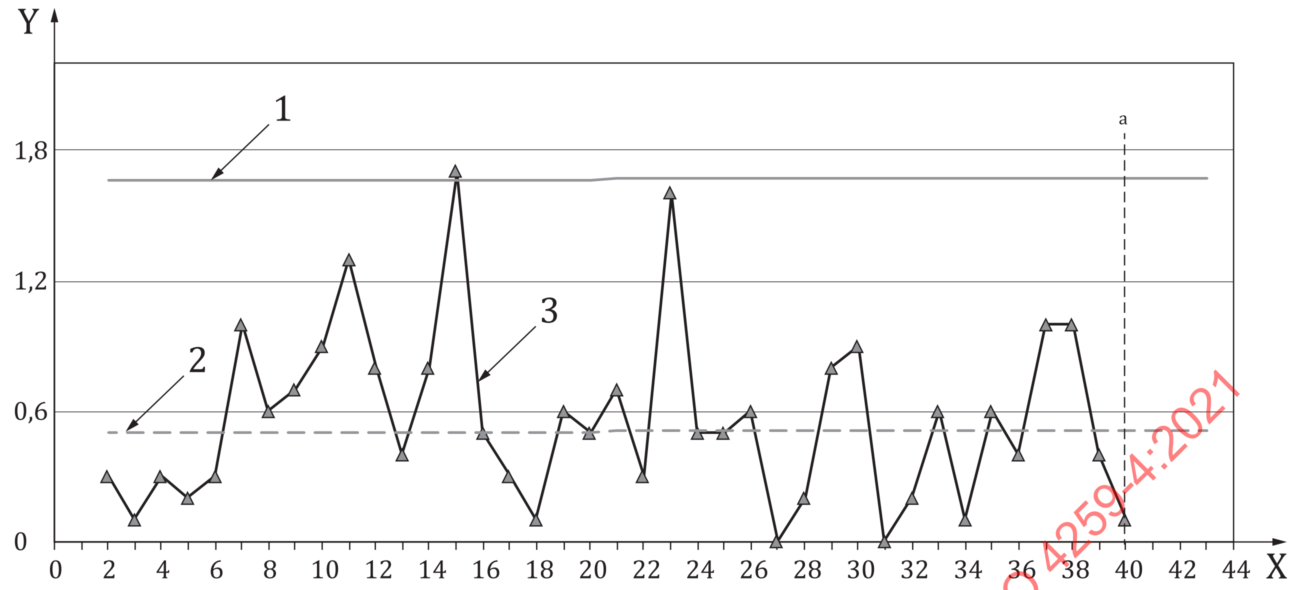
Figure A.6 — I chart stage 2 generated

Table A.7 — Data for Figures A.6 and A.7

Obs. No. (i)	Test result (X)	EWMA	$\bar{x}$	UCL X	LCL X	UCL EWMA	LCL EWMA	MR	$\overline{MR}$	UCL MR
1	6,7	6,93	7,08	8,89	5,26	7,98	6,17			
2	7	6,96	7,08	8,89	5,26	7,98	6,17	0,3	0,51	1,67
3	6,9	6,93	7,08	8,89	5,26	7,98	6,17	0,1	0,51	1,67
4	6,6	6,80	7,08	8,89	5,26	7,98	6,17	0,3	0,51	1,67

Table A.7 *(continued)*

Obs. No. (i)	Test result (X)	EWMA	$\bar{x}$	UCL X	LCL X	UCL EWMA	LCL EWMA	MR	$\overline{MR}$	UCL MR
5	6,8	6,80	7,08	8,89	5,26	7,98	6,17	0,2	0,51	1,67
6	7,1	6,92	7,08	8,89	5,26	7,98	6,17	0,3	0,51	1,67
7	8,1	7,39	7,08	8,89	5,26	7,98	6,17	1	0,51	1,67
8	7,5	7,44	7,08	8,89	5,26	7,98	6,17	0,6	0,51	1,67
9	6,8	7,18	7,08	8,89	5,26	7,98	6,17	0,7	0,51	1,67
10	7,7	7,39	7,08	8,89	5,26	7,98	6,17	0,9	0,51	1,67
11	6,4	6,99	7,08	8,89	5,26	7,98	6,17	1,3	0,51	1,67
12	7,2	7,08	7,08	8,89	5,26	7,98	6,17	0,8	0,51	1,67
13	6,8	6,97	7,08	8,89	5,26	7,98	6,17	0,4	0,51	1,67
14	6,0	6,58	7,08	8,89	5,26	7,98	6,17	0,8	0,51	1,67
15	7,7	7,03	7,08	8,89	5,26	7,98	6,17	1,7	0,51	1,67
16	7,2	7,10	7,08	8,89	5,26	7,98	6,17	0,5	0,51	1,67
17	6,9	7,02	7,08	8,89	5,26	7,98	6,17	0,3	0,51	1,67
18	6,8	6,93	7,08	8,89	5,26	7,98	6,17	0,1	0,51	1,67
19	7,4	7,12	7,08	8,89	5,26	7,98	6,17	0,6	0,51	1,67
20	7,9	7,43	7,08	8,89	5,26	7,98	6,17	0,5	0,51	1,67
21	7,2	7,34	7,08	8,89	5,26	7,98	6,17	0,7	0,51	1,67
22	6,9	7,16	7,08	8,89	5,26	7,98	6,17	0,3	0,51	1,67
23	8,5	7,70	7,08	8,89	5,26	7,98	6,17	1,6	0,51	1,67
24	8,0	7,82	7,08	8,89	5,26	7,98	6,17	0,5	0,51	1,67
25	7,5	7,69	7,08	8,89	5,26	7,98	6,17	0,5	0,51	1,67
26	6,9	7,37	7,08	8,89	5,26	7,98	6,17	0,6	0,51	1,67
27	6,9	7,18	7,08	8,89	5,26	7,98	6,17	0	0,51	1,67
28	7,1	7,15	7,08	8,89	5,26	7,98	6,17	0,2	0,51	1,67
29	6,3	6,81	7,08	8,89	5,26	7,98	6,17	0,8	0,51	1,67
30	7,2	6,97	7,08	8,89	5,26	7,98	6,17	0,9	0,51	1,67
31	7,2	7,06	7,08	8,89	5,26	7,98	6,17	0	0,51	1,67
32	7,4	7,20	7,08	8,89	5,26	7,98	6,17	0,2	0,51	1,67
33	6,8	7,04	7,08	8,89	5,26	7,98	6,17	0,6	0,51	1,67
34	6,9	6,98	7,08	8,89	5,26	7,98	6,17	0,1	0,51	1,67
35	7,5	7,19	7,08	8,89	5,26	7,98	6,17	0,6	0,51	1,67
36	7,1	7,15	7,08	8,89	5,26	7,98	6,17	0,4	0,51	1,67
37	6,1	6,73	7,08	8,89	5,26	7,98	6,17	1	0,51	1,67
38	7,1	6,88	7,08	8,89	5,26	7,98	6,17	1	0,51	1,67
39	7,5	7,13	7,08	8,89	5,26	7,98	6,17	0,4	0,51	1,67
40	7,6	7,32	7,08	8,89	5,26	7,98	6,17	0,1	0,51	1,67
41			7,13	8,91	5,35	8,02	6,24		0,51	1,67
42			7,13	8,91	5,35	8,02	6,24		0,51	1,67
43			7,13	8,91	5,35	8,02	6,24		0,51	1,67



Key

X	observations	upper line	1	$UCL\_\overline{MR} = 3{,}27\,\overline{MR}$
Y	*MR*	dashed line	2	$\overline{MR} = 0{,}51$
		triangled-line	3	MR results

a Revised limit applies to chart.

Figure A.7 — MR chart generated

A.2.2 Scenario 2 (see 4.3.3.2.3)

Archiving control charts and updating s_{known}, df_s_{known}, $\overline{\overline{MR}}_{known}$.

"When it is necessary to change the I-chart QC material, either due to run out of QC material, or operational reasons, compute the final achieved average $\bar{x}_{achieved}$, standard deviation $s_{achieved}$, $df\_s_{achieved}$, $\overline{\overline{MR}}_{achieved}$, $n_{achieved}$ using all in-statistical-control data for the specific batch of QC material. Designate $s_{achieved}$ as the current s_{known} and $\overline{\overline{MR}}_{achieved}$ as the $\overline{\overline{MR}}_{known}$ associated with the current s_{known} for this type of QC material. Archive (retire) the control chart".

The QC batch for this example is exhausted after the 55th result.

A final set of statistics is computed using all in control data for the entire chart.

$\bar{x}_{achieved} = 7{,}162$; $s_{achieved} = 0{,}511$; $\overline{\overline{MR}}_{achieved} = 0{,}565$; $n_{achieved} = 55$

$df\_s_{achieved} = n_{achieved} - 1 = 54$

current s_{known} is updated from 0,623 (at Stage 1 start) to $s_{achieved}$ = 0,511;

associated $\overline{\overline{MR}}_{known}$ is updated from 0,487 (at Stage 1 start) to $\overline{\overline{MR}}_{achieved}$ = 0,565

At start of Stage 1 for this archived control chart:

$s_{known} = 0{,}623$ with working range: $\bar{x}$ from 7,132 to 7,305; $\overline{\overline{MR}}_{known} = 0{,}487$ $df\_s_{known} = 75$

From 4.3.3.2.3 Scenario 2:

“if s_{stage1} for this to-be-archived control chart is obtained by pooling with an existing s_{known}, add $\bar{x}_{achieved}$ to the working range associated with this latest s_{known}; increase the df_s_{known} by adding $df\_s_{achieved}$ to the df_s_{known} for the previously existing df_s_{known}.

Since $\bar{x}_{achieved}$ = 7,162 is within the working range for previous s_{known} (7,132 to 7,305), the working range of current s_{known} does not need to be updated; df_s_{known} is updated by adding 54 to 75 = 129

A.3 QC Material batch change transition (see 4.4)

A.3.1 Procedure 2, Q-chart (see 4.4.3)

The formula for the Q statistic is reproduced from Case II of Reference [6]:

$$Q_r = \sqrt{\frac{(r-1)}{r}} \left(\frac{(x_r - \bar{x}_{r-1})}{\sigma_0} \right) \text{ for } r = 2, 3, \ldots\ldots \qquad \text{(A.5)}$$

where

- Q_r is the the Q statistic computed using the latest (r^{th}) test result x_r;
- r is the the chronological number for the result as it arrives in time;
- $\bar{x}_{r-1}$ is the the average calculated using all past results up to r – 1;
- σ_0 is s_{known} for the new batch of QC material type.

The Q_r statistic has no associated measurement units and can be plotted on a standardised I- control chart with fixed, forward looking control limits computed using $\bar{x}$ = 0 and s = 1. This will be referred to as the Q-chart.

Continuing with the example above, a single test of the new batch of QC material is 7,8. An accompanying test of a check standard with ARV = 7,8 is 8,3. The current s_{known} at the nominal ARV level is 0,511 (from the archived control chart). Since 8,3 is within the ARV ± 1,5s_{known}, the new QC result of 7,8 is considered to be validated.

The Q-chart for the next 19 results is shown in Figure A.8.

